

Morphological study of silver sulfide layers on the polypropylene surface

Valentina Krylova^{1*},

Rasa Alaburdaitė²,

Asta Guobienė³

¹ Department of Inorganic Chemistry,
Kaunas University of Technology,
Radvilėnų Str. 19, LT-50254 Kaunas

² Department of General Chemistry,
Kaunas University of Technology,
Radvilėnų Str. 19, LT-50254 Kaunas

³ Institute of Materials Science,
Kaunas University of Technology,
Savanorių Ave. 271,
LT-50131 Kaunas, Lithuania

Layers of silver sulfide, Ag₂S, on the surface of polypropylene (PP) film were formed by the chemical bath deposition method (CBD). An initial treatment of PP with modifiers is required in order to provide the adherence to the polymer surface. Film samples were characterized by X-ray diffractometry (XRD), optical microscopy, scanning electron microscopy (SEM) / energy dispersive X-Ray (EDX) analysis and atomic force microscopy (AFM). XRD and EDX methods confirmed that on the surface of the polypropylene film a layer of silver sulfide was formed. The surface morphology, texture and uniformity of the silver sulfide layers formed on the PP surface depend on the number of polymer immersions into the precursor solution. The formation of silver sulfide layers proceeded irregularly in the form of islands which grew into larger agglomerates.

Key words: Ag₂S, polypropylene, surface modification, XRD, SEM, AFM

INTRODUCTION

Recently, the modification of polymeric surfaces by formation of a thin layer of inorganic compounds has attracted an increasing interest. This is because these composite materials possess new, more intensive characteristic properties, such as wettability, mechanical adhesion, dyeing, electrical, optical, and other properties, which allow applying them in various fields of science and technology [1–4]. Among these polymers, polypropylene (PP) is a promising polymer due to its facile processing and low cost. The plastic industry demands great amounts of PP (mainly for packaging), however, it is also used in layers with paper, wood, and other plastics [5].

PP films are used for membrane manufacturing which is needed for ultra- and nanofiltration [2].

Polypropylene is resistant to many solvents and chemicals. It is known by its apolar characteristic, which has direct influence on its adhesion properties. Surface free energy of a solid surface gives a direct measure of intermolecular interactions at interfaces and has a strong influence on adsorption, adhesion behaviour, and surface roughness [3, 4, 6, 7]. One of the modification methods for the polypropylene surface is oxidation [1–3, 8–12]. The oxidation of PP in the solid phase is done in the reactions with ozone, γ -initiated, corona method, and UV ray or with chemical oxidizing mixture [3]. Oxidation potential of PP is greatly enhanced due to the presence of tertiary carbons and unsaturated sites in PP. The oxidation of PP leads to the formation of oxygen-containing

* Corresponding author. E-mail: Valentina.Krylova@ktu.lt

groups, which greatly affect the surface polarity and the adhesion properties of the polymer.

A lot of chemical and physical methods can be used in the modification process of the polymer surface with the layers of conductive and semiconductive metal compounds: vacuum evaporation, spray pyrolysis, physical and plasma enhanced chemical vapour deposition, electroless deposition, successive ionic layer adsorption and reaction, chemical bath deposition, etc. [1, 2, 5, 13–17].

The thin semiconductive silver sulfide films have a number of applications in various devices, such as solar cells, photoconductors, IR detectors, ion selective electrodes, where adhesion, optical and semiconductor [18–24] properties are needed. Metal sulfide layer on polyolefin's surface improves mechanical adhesion of the polymer and, thus, these polymeric matrices can be used for metallic covering [25].

In this study, we synthesized Ag_2S thin film using a simple, cost effective, and highly reproducible technique – the chemical bath deposition (CBD) method. The technology is based on slow controlled precipitation of the desired compound from its ions in the reaction bath solution. A complexing agent is usually employed to control the reaction in a suitable medium as indicated by the pH to obtain crystal growth.

The aim of the present work was to form and characterize surface morphology of silver sulfide layers on the surface of the hydrophobic polymeric material – polypropylene (PP) film (PP/ Ag_2S) by the means of optical microscopy, Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM, EDS), and to determine the influence of the number of immersions in aqueous solutions of silver nitrate (AgNO_3)/sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) on morphologic characteristics.

EXPERIMENTAL

15 mm × 70 mm size samples of non-oriented isotactic polypropylene (PP) film of 150 μm thicknesses (Proline X998, KWH Plast, Finland) were used for the experiments.

Before Ag_2S formation process, the hydrophobic PP sample requires an initial surface treatment process in order to facilitate its adhesion properties. Polymer was firstly treated for 25 min at 90 °C with oxidizing solution ($\text{H}_2\text{SO}_4/\text{H}_3\text{PO}_4$ (1 : 1), saturated with CrO_3). The formation of Ag_2S layers on PP was carried out in a glass reactor. Oxidized PP films were immersed for 40 min in aqueous $\text{Na}_2\text{S}_2\text{O}_3$ (0.2 M) and acidified with pH 2.3 AgNO_3 (0.06 M) solutions at 20 °C. The obtained samples were removed from the reaction solution, rinsed with distilled water, and dried at room temperature. The Ag_2S /PP samples were subjected to repetitive immersions in order to increase the Ag_2S amount. After that, the Ag_2S /PP samples were repeatedly immersed in the freshly prepared reaction solution. The immersions procedure was repeated 5 times.

Distilled water and analytically pure $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, AgNO_3 , HNO_3 reagents were used to prepare reaction solutions.

Solutions' pH was measured by a pH-meter WTW330, with a combinative glass and Ag/AgCl electrode and a temperature meter WTW SenTix 41 (Germany).

X-ray diffractometry was carried out under a Bragg-Brendan circuit on a diffractometer (Dron-6, Russia) using $\text{Cu K}\alpha$ ($\lambda = 0.154178$ nm) radiation, 30 kV voltage and 30 μA current. The scanning range was $2\theta = 23\text{--}60^\circ$. The scanning speed was $1^\circ/\text{min}$. Results were registered in *in situ* mode with a computer, and X-ray diffractograms of the samples were treated using the Search Match, Xfit, ConvX, Dplot95, and Photo Styler programs to eliminate the PP peaks.

Optical microscopy of PP samples modified by silver sulfide was carried out by an optical microscope Olympus CX31 (Olympus, Philippines) and a photo camera Olympus C-5050 (Olympus, Japan, ×400).

The morphology of the surface of Ag_2S layers and the quantitative microscopy of roughness of the formed layers on the surface of PP were studied with a NT-206 atomic force microscope (Belarus), in the contact regime with high resolution probes with the force constant $k = 3$ N/m. The characteristics of the atomic force microscope: the maximum scan field area from 12×12 up to 30×30 microns, the measurement matrix up to 512×512 points and more, the maximum range of measured heights 4 microns, lateral resolution 2 nm, and vertical resolution 0.1–0.2 nm.

The scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS) Quantax 200 with a detector X Flash 4030 (Bruker AXS Microanalysis GmbH, Germany) was applied for analysis of the surface of the obtained Ag_2S layers.

RESULTS AND DISCUSSION

In order to obtain Ag_2S layers on the PP film it was necessary to find the optimal conditions of the chemical bath deposition process. According to the bibliography, the impregnation of CuS on polyethylene [4] or polyimide [12] requires a pretreatment process in order to facilitate its adhesion. It has been determined that the optimal pretreatment temperature for PP of 150 μm thickness is 90 °C and the optimal duration is 25 min in the solution of $\text{H}_2\text{SO}_4/\text{H}_3\text{PO}_4$ (1 : 1) saturated with CrO_3 [26].

The attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectrum of the pretreated PP presented previously [27] has shown that polypropylene characteristic peaks sharply diminish and a new signal appears at 1719 cm^{-1} . This signal is probably due to superposition of signals at 1710 cm^{-1} and 1724 cm^{-1} , characteristic of carbonylic and carboxylic stretching vibrations, respectively. The spectrum of the pretreated PP confirms that the polypropylene surface has been oxidized.

The silver sulfide layers may be deposited on the PP films by immersing the polymer in aqueous solutions of silver nitrate (AgNO_3) and sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). When a soluble sodium thiosulfate reacts with silver nitrate, insoluble

silver thiosulfate, $\text{Ag}_2\text{S}_2\text{O}_3$, is formed. $\text{Ag}_2\text{S}_2\text{O}_3$ is comparatively unstable and hydrolyzes to release Ag_2S into the reaction solution.

The initial experiments indicated that the usage of 0.1–0.2 M acidified AgNO_3 and 0.1–0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions for Ag_2S layers depositing on PP resulted in quick sedimentation of Ag_2S precipitates on the reactor bottom and layers on polypropylene could not be formed. Therefore, slow precipitation of silver sulfide particles is essential for the formation of the layers on the polymer surface.

In order to diminish the rate of Ag_2S formation process, the silver ions were complexed by thiosulfate ions. When the $\text{Na}_2\text{S}_2\text{O}_3$ solution was added into the aqueous AgNO_3 solution, silver ions were coordinating with some thiosulfate groups, resulting in a relatively high concentration of silver ions around these groups. In the excess of thiosulfate, the soluble mononuclear $[\text{Ag}(\text{S}_2\text{O}_3)_n]^{(2n-1)-}$ and polynuclear $[\text{Ag}_2(\text{S}_2\text{O}_3)_2]^{2-}$, $[\text{Ag}_2(\text{S}_2\text{O}_3)_3]^{4-}$, $[\text{Ag}_3(\text{S}_2\text{O}_3)_5]^{7-}$ [28, 29] complexes were formed.

In the nitric acid solution these complexes decomposed and slowly released the S^{2-} ions combined with the Ag^+ ions to form a very insoluble Ag_2S nucleus on some special sites of PP.

It has been found that the 0.06 M acidified AgNO_3 and 0.20 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions are the most suitable for the formation of Ag_2S layers on the PP surface at pH 2.3 and 20 °C. In the previous work [30] it was determined that Ag_2S particles have a negative charge of the zeta potential (approximately -47 – -54 mV) and it was reflected by the stability of suspensions which showed little aggregation upon standing for 40 min.

The deposited layers were subjected to X-ray diffractometry to investigate the crystallographic structure of these layers. Structural studies of the deposits on polypropylene are limited by the crystallinity of the PP film itself. The intensity of its peaks exceeds the intensity of silver sulfide peaks several times in $2\theta \leq 23^\circ$. Therefore, the area of $2\theta \geq 23^\circ$ was investigated in more detail.

Fig. 1 presents the X-ray diffraction pattern of the oxidized PP (Fig. 1, curve 1) and as-prepared $\text{Ag}_2\text{S}/\text{PP}$ composites (Fig. 1, curves 2 and 3). After the first immersion in the precursor solution, the diffraction pattern of deposits on the polypropylene sample gave dominant peaks 2θ at 26.19°, 29.06°, and 43.47° (Fig. 1, curve 2), and after 5 immersions more peaks at 2θ appeared: 31.82°, 33.66°, 34.74°, 36.82°, 37.66°, and 46.28° (Fig. 1, curve 3). These peaks show the polycrystalline nature of the prepared layer. The diffraction patterns were indexed to the monoclinic Ag_2S phase and are in good agreement with the reported data for $\alpha\text{-Ag}_2\text{S}$ (acanthite) (JCPDS Card File: 00-014-0072).

The changes on the surface structure were monitored by optical microscopy. Images of the PP sample surfaces ($\times 400$) showed that the structure of the oxidized PP surface revealed a concentric internal structure with gaps and cavities (Fig. 2a). Images showed that deeper grains were not treated by oxidizing solution.

The optical micrographs of the PP/ Ag_2S films after the first and fifth immersions in the precursor solution are shown in Fig. 2b and 2c. PP surface morphology changed and some irregularities appeared after the first treatment immersions (Fig. 2b) and they showed the nucleation process and

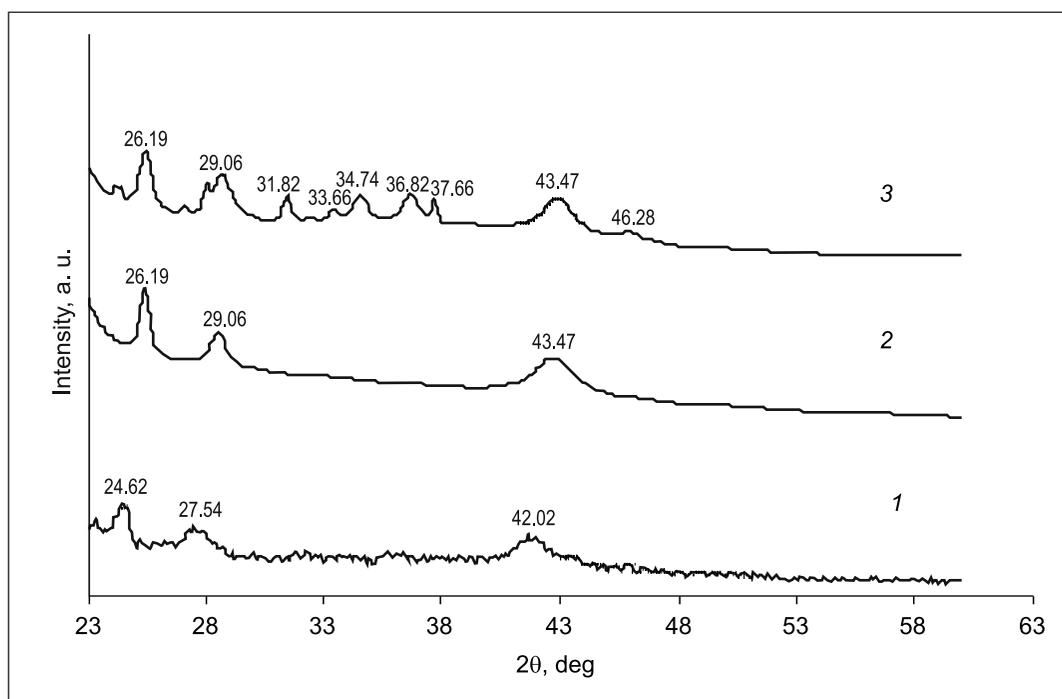


Fig. 1. X-ray diffractograms of the Ag_2S layers formed on oxidized PP samples immersed in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C for different immersions time, min: 1 – 0; 2 – 40; 3 – 200 (5×40 min)

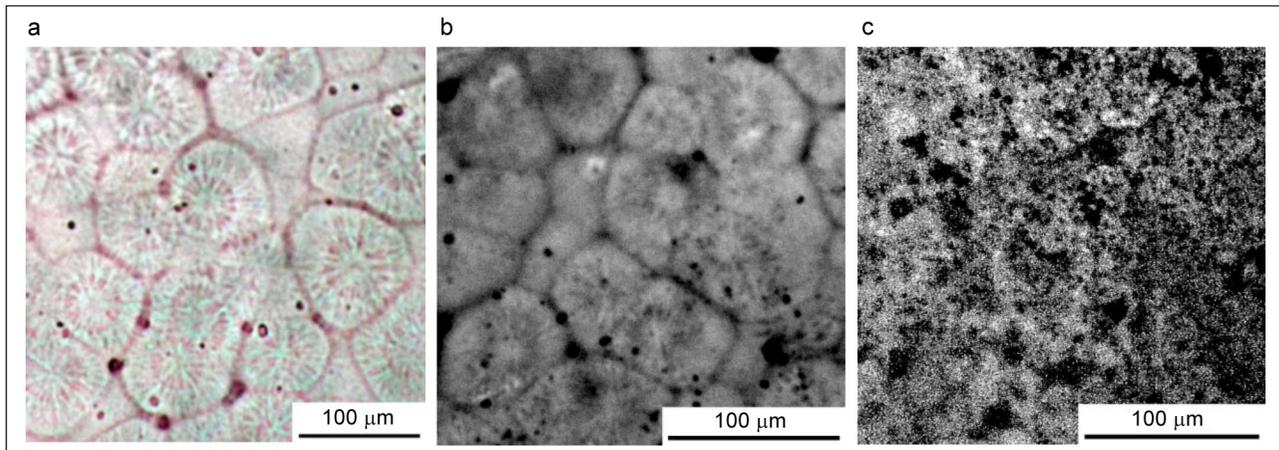


Fig. 2. Micrographs of oxidized PP samples ($\times 400$) immersed in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C for different immersions time, min: a – 0; b – 40; c – 200 (5×40 min)

growing of the Ag_2S nucleus; the growing nucleus spread in all directions and began the formation of new agglomerates over the first formed layer and these latter particles spread in all directions, occupying the uncovered surface and / or the previous layer. Fig. 2c displays the view of modified PP surface after the 5th immersion in the precursor solution. It is possible to observe regions with good agglomeration and the fusion of small dendrites. After the 5th immersion the silver sulfide layer became more compact because the formed Ag_2S layer was thicker.

Lateral force microscopy studies are useful for imaging variations in surface friction that can arise from inhomogeneity in surface material, and also for obtaining edge-enhanced images.

Fig. 3a shows the representative SEM images of the Ag_2S layers on the PP surface formed after the 5th immersion (5×40 min) of the polymer in the precursor solution. The silver sulfide surface exhibits non-homogeneous morphology. The irregularly shaped grains join to form different size islands on PP surfaces. The maximum size of islands in the scanned area is $21.7 \times 29 \mu\text{m}$. The large number of micro bumps and irregularly spread clusters are also visible.

The thickness of the Ag_2S layer was evaluated from the cross-sectional scanning electron microscopy photographs (Fig. 3b). Ag_2S formed during PP treatment in the precursor solution was deposited on the polymer surface and layer which thickness after the 5th immersion with duration of 200 min (5×40 min) was about $12 \mu\text{m}$.

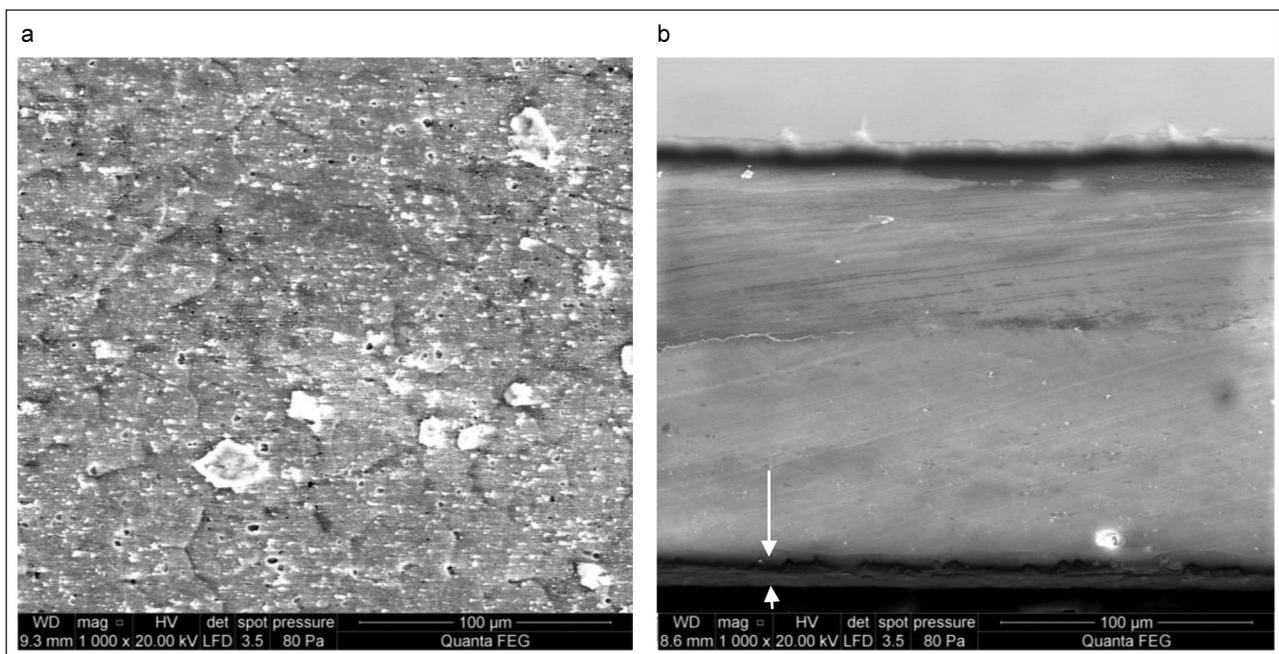


Fig. 3. SEM (a) and cross-sectional (b) micrographs of the oxidized PP surface immersed for 5 times for 200 min (5×40 min) in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C

The elemental composition of the formed layers on PP was evaluated by EDX. It showed (Fig. 4) the presence of silver (Ag) and sulfur (S) in the coatings.

The samples of the oxidized PP were immersed in the precursor solution several times in order to study the morphology characteristics of the surface according to the incorporated Ag_2S amount. The immersion steps were accompanied by atomic force microscopy. Fields of 12×12 microns were investigated; for the quantitative estimation of the surface, the standard programs “Surface Scan” and “Surface Explorer” of the view treatment were used.

A view of the oxidized PP surface is presented in Figs. 5–6 and shows a continuous roughness distribution. The oxidized

PP surface (Fig. 5) was rough with a mean roughness R_q slightly greater than 15 nm, the skewness was ~ 0.19 nm. The height of PP grains was ~ 80 nm and diameter was ~ 0.3 μm .

It was possible to determine the average height and diameter of the grains from the cross sections of Ag_2S layers formed on the PP surface when the polymer was immersed in precursor solutions for one and five times. When the polymer was immersed once (40 min) in the precursor solution, the height of the grain composite (PP and Ag_2S) reached 170 nm and the diameter was ~ 0.5 μm (Figs. 7–8). The surface mean roughness of the composite was 38 nm. When the polymer was treated in the solution for a short duration, the silver sulfide layer formed an island on the PP surface.

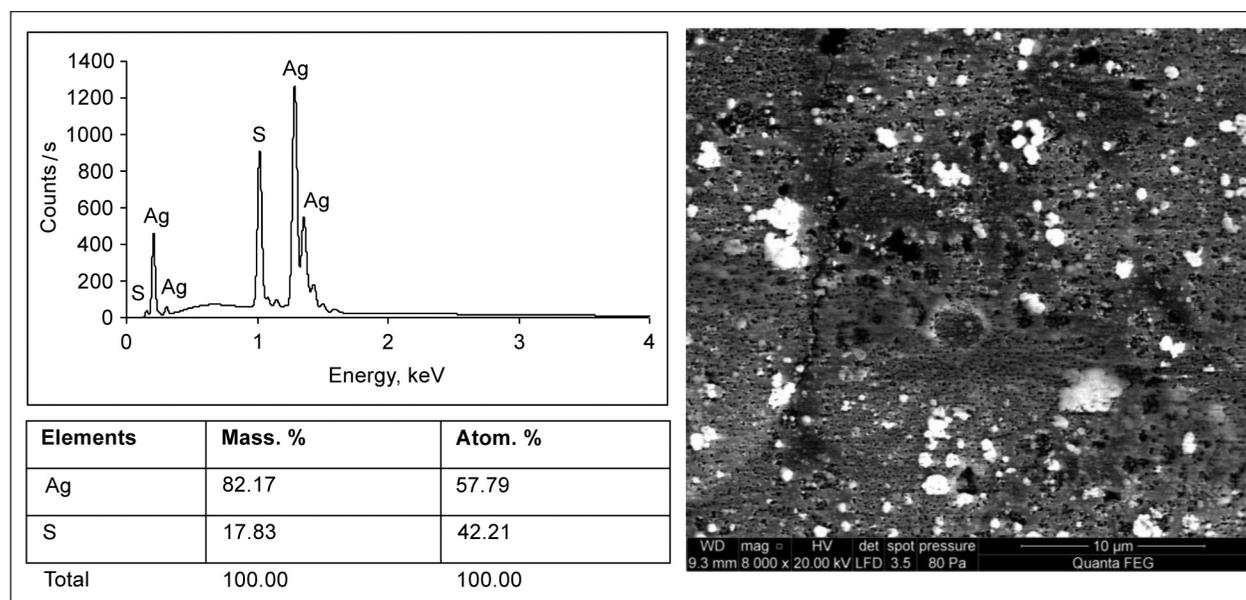


Fig. 4. Elemental composition of the deposit by SEM with an elemental EDX analyser of oxidized PP immersed for 5 times for 200 min (5×40 min) in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C

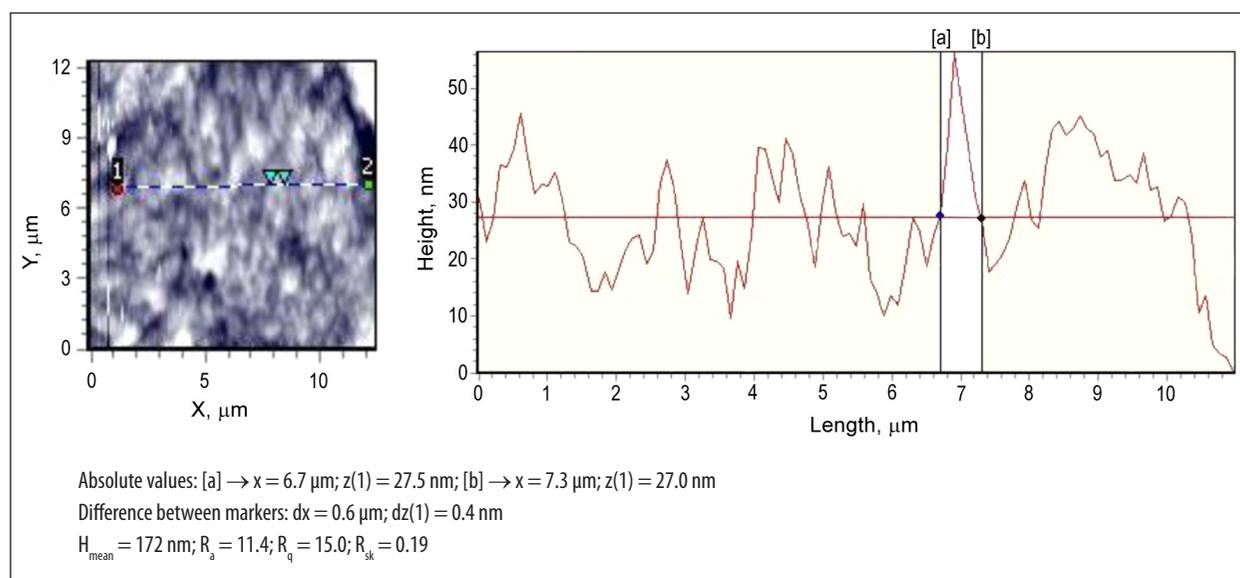


Fig. 5. A view of the topographic image and a line profile of the oxidized for 25 min at 90 °C PP surface

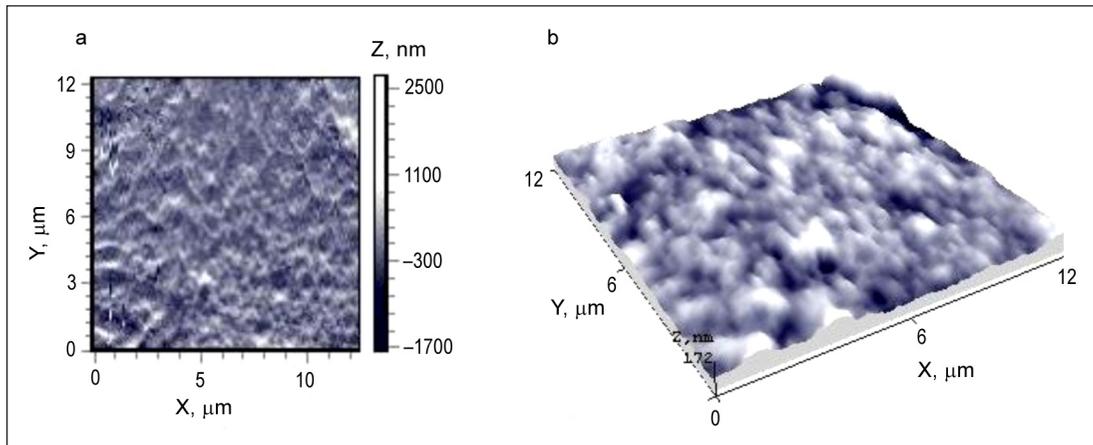


Fig. 6. An atomic force microscopy view of the oxidized PP surface: a 2D view of lateral forces microscopy (a) and a 3D topography view (b)

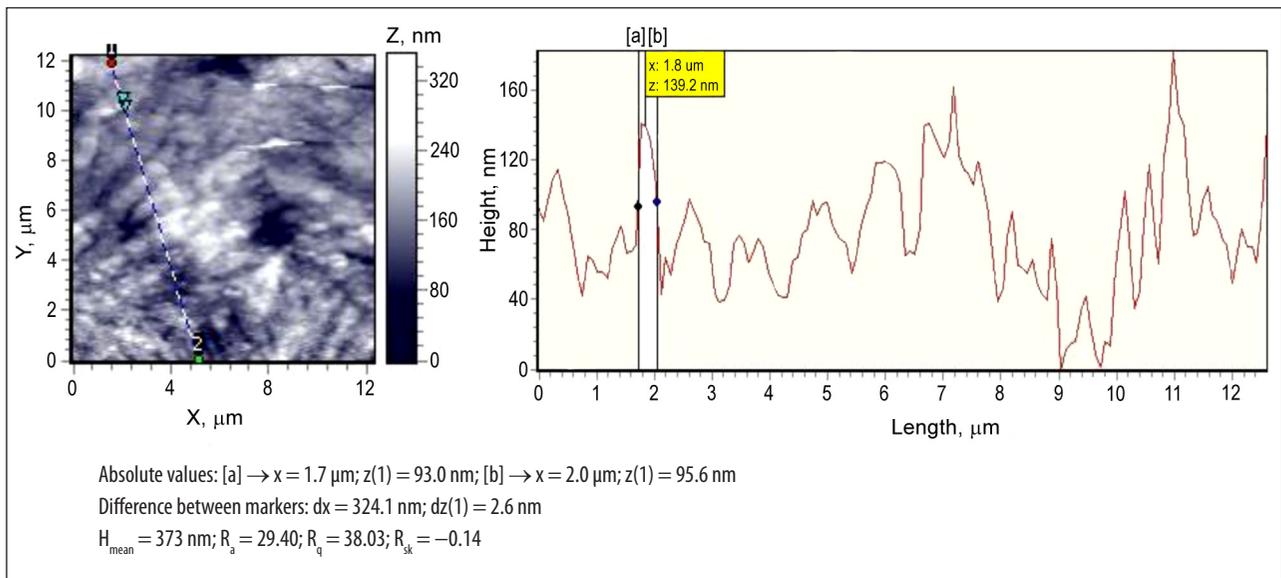


Fig. 7. A view of the topographic image and a line profile of the silver sulfide layer formed on the oxidized PP surface. PP was immersed 1 time for 40 min in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C

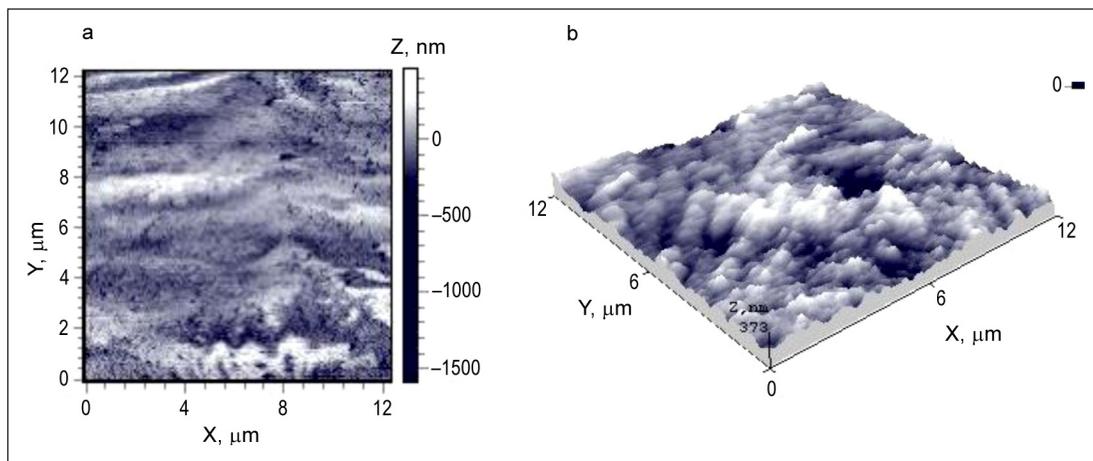


Fig. 8. An atomic force microscopy view of Ag_2S layers formed on the oxidized PP surface: a 2D view of lateral forces microscopy (a) and a 3D topography view (b). PP was immersed 1 time for 40 min in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C

When the number of immersions increased, the growth of the silver sulfide layer proceeded more intensively. We observed an island aggregation process. When the number of immersions was 5, the height of the grains increased to 600 nm and the diameter was $\sim 0.7 \mu\text{m}$, and the mean roughness reached 74 nm (Figs. 9–10).

The size of grains, like the roughness of the layer, was increasing with increasing the number of immersions of the oxidized PP in the precursor solution (Fig. 11). We observed the island (3D) growth.

The oxidized PP skewness (R_{sk}) was equal to 0.19 nm and indicated that raised elements dominated on the surface. After the first immersion of PP samples, R_{sk} was equal to -0.14 nm and indicated that the surface became porous, deep elements dominated. When the number of immersions increased to five, skewness became equal to -0.06 nm , which indicated that deep elements still dominated in the surface, however, porosity decreased. The asymmetry parameter R_{sk}

displayed the domination of islands, grains, and agglomerates on the surface.

Dependence of surface morphology of the silver sulfide layers formed on the oxidized PP surface on the number of immersions of the polymer in the precursor solution was determined. During the 1st immersion of the oxidized PP in the $\text{AgNO}_3/\text{Na}_2\text{S}_2\text{O}_3$ solution, the PP and Ag_2S polycomposite formed. It was assumed that the PP surface was filled with Ag_2S particles. The next immersion of the oxidized PP in the precursor solution induced the Ag_2S island growth. We found that the composition and uniformity of the layer depended on the number of immersions.

The data about formation of the Ag_2S layer on the polyamide (PA) surface were presented previously [30]. Silver sulfide layers formed on the PP surface differ from such layers on the PA surface because the Ag_2S layers are deposited on polymers with different surface morphological parameters. In comparison, the root mean square roughness of virgin PA

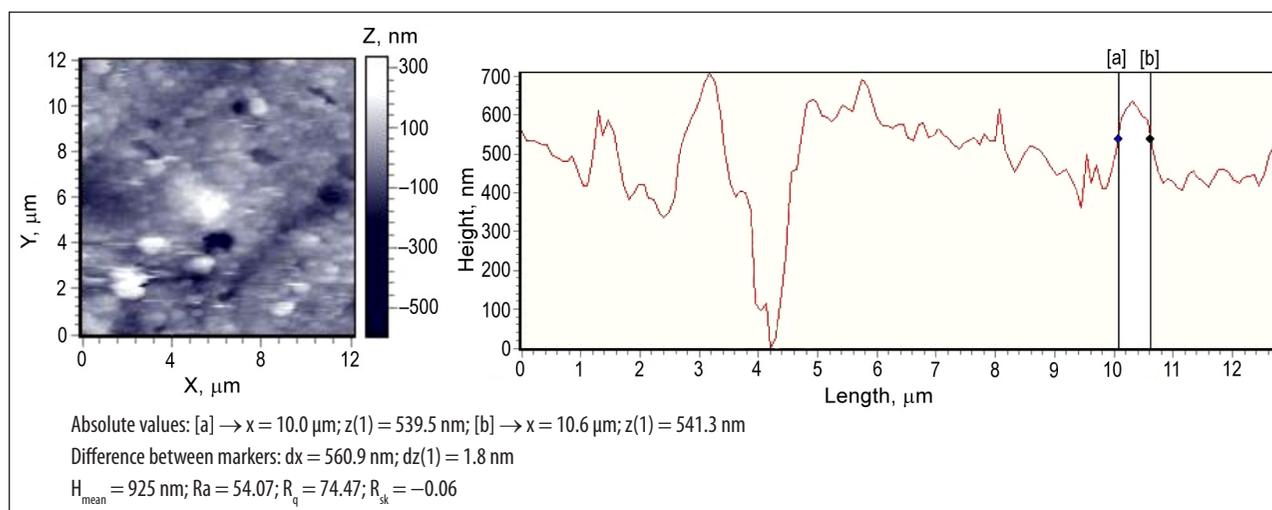


Fig. 9. A view of the topographic image and a line profile of the silver sulfide layer formed on the oxidized PP surface. PP was immersed 5 times for 200 min ($5 \times 40 \text{ min}$) in acidified aqueous 0.06 M AgNO_3 and $0.2 \text{ M Na}_2\text{S}_2\text{O}_3$ solutions at 20°C

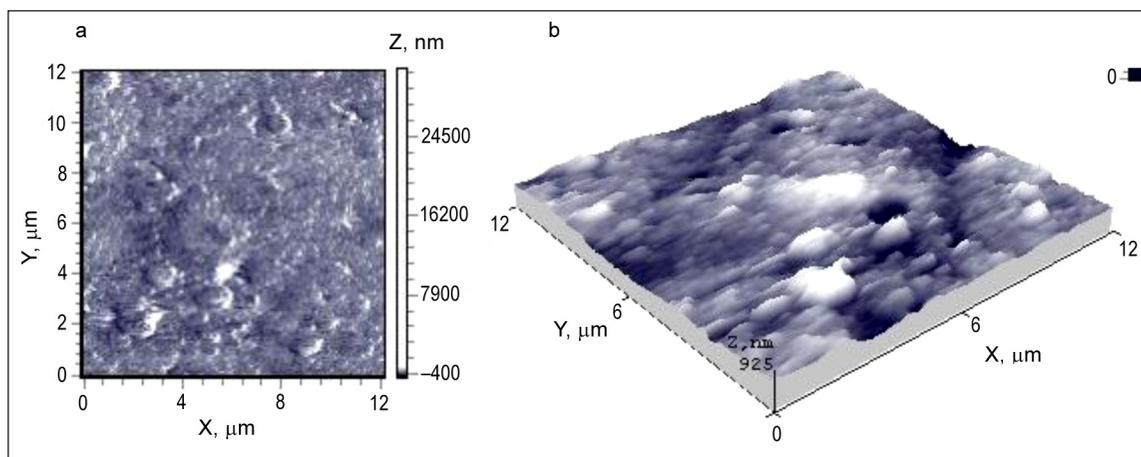


Fig. 10. An atomic force microscopy view of Ag_2S layers formed on the oxidized PP surface: a 2D view of lateral forces microscopy (a) and a 3D topography view (b). PP was immersed 5 times for 200 min ($5 \times 40 \text{ min}$) in acidified aqueous 0.06 M AgNO_3 and $0.2 \text{ M Na}_2\text{S}_2\text{O}_3$ solutions at 20°C

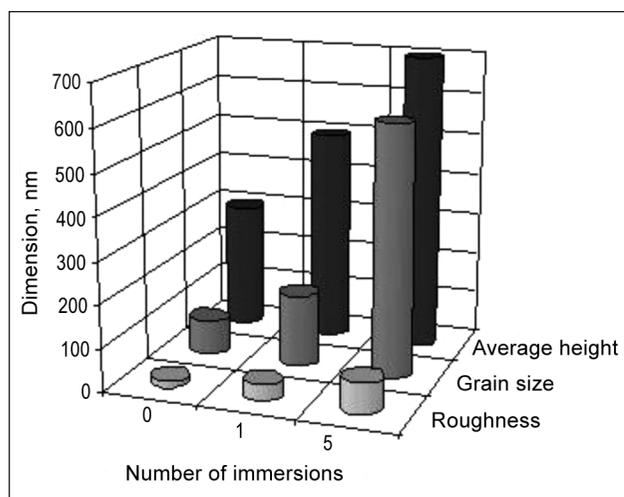


Fig. 11. Dependence of the layer mean square roughness (R_q), average mean height (H_{mean}) and diameter of grain on the number of immersions of oxidized PP in acidified aqueous 0.06 M AgNO_3 and 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solutions at 20 °C

(R_q 4.6 nm) was ~3 times lower than that of the oxidized PP surface (R_q 15 nm); the skewness was 4 times higher (~0.8 nm) than that of the oxidized PP (~0.19 nm). Other morphological parameters of PA are as follows: the height of grains was ~100 nm and the diameter was ~788 nm. The height of oxidized PP grains was ~80 nm and the diameter was ~0.3 μm . We can compare variation of the main morphological parameters of deposited Ag_2S layers on these polymers: after the first PA immersion in the precursor solution, the average height of the composite reached 145 nm, and the one on PP was 3.5 times higher (488 nm), the layer roughness was nearly equal to 32 nm and 38 nm, respectively. When the number of immersions was 5, the average height of the composite on polyamide increased to 1 093 nm, and the one on PP grew only to 690 nm, i. e. it was lower ~1.6 times; the roughness increased to ~244 nm, and the one on PP was only 74 nm. It was assumed that the Ag_2S layers formation process on the PP surface was more uniform.

In our work [27] the Ag_2S layer on polypropylene was characterized as an Ag^+ ion selective electrode in terms of potential response and detection limit. The electrode was also tested as an end-point electrode for argentometric titration of vitamin B_1 (thiamine hydrochloride). It was established that the PP/ Ag_2S sensor exhibited a low detection limit and therefore it is promising as an indicating electrode in argentometric titration of vitamin B_1 as well as a sensor for determination of silver ions.

CONCLUSIONS

1. The XRD studies indicated that the deposited layers were polycrystalline with a monoclinic (acanthite) crystal structure.

2. Optical micrographs have shown the uniform structure present in the oxidized PP film. The Ag_2S layer formed on PP became more compact after the 5th immersion.

3. SEM/EDX analyses confirmed that deposition of the Ag_2S coating took place on the surface of PP.

4. A cross-sectional SEM microphotograph of the PP/ Ag_2S film has shown that the thickness of layers depends on the number of immersions in the precursor solution and after 5 immersions it was about 12 μm .

5. The Ag_2S film growth starts only on some spots on the substrate surface. After the total coverage of the substrate is achieved, the film gets smoother; it is homogeneous and rather flat.

6. The surface morphology, contexture and uniformity of the silver sulfide layers formed on the oxidized PP surface depend on the number of immersions of the polymer in the precursor solution.

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SIDABRO SULFIDO SLUOKSNIŲ POLIPROPENO PAVIRŠIUJE MORFOLOGINIAI TYRIMAI

S a n t r a u k a

Straipsnyje aptarta organinio polimerinio paviršiaus ir neorganinių junginių sąveika, kuriai būtinas pirminis polimero paviršiaus išdėdymas siekiant pagerinti jo adheziją. Rentgenostruktūrinės ir rentgeno spindulių energijos dispersinės analizės metodais nustatyta, kad polipropeno plėvelių paviršiuje cheminio nusodinimo būdu suformuoti sidabro sulfido sluoksniai, kurių paviršius ištirtas optiniu mikroskopu, skenuojančiu elektroniniu mikroskopu ir atominių jėgų mikroskopu. Nustatytas sidabro sulfido sluoksnių formavimosi mechanizmas, paviršiaus šiurkštis, sluoksnių storis.